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Recovery of Lithium From a Montmorillonite-Type Clay

By R. H. Lien

UNITED STATES DEPARTMENT OF THE INTERIOR

BUREAU OF MINES 75TH ANNIVERSARY

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By R. H. Lien

With an Appendix on Process Economics by D. A. Kramer

UNITED STATES DEPARTMENT OF THE INTERIOR
Donald Paul Hodel, Secretary

BUREAU OF MINES
Robert C. Horton, Director

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CONTENTS

	Page
Abstract	1
Introduction	2
Process description	3
Materials	4
PRU equipment	5
Operating conditions and test results	9
Feed preparation	9
Roasting	9
Roasting mechanism	9
Batch tests	9
Continuous roast tests	10
Leaching	11
Solids content and wash water recycle	11
Calcine particle size and leach time	11
Summary of leach test results	12
Evaporation	12
Product precipitation	13
Crystallization	13
Overall lithium recovery	14
Material balance and economic evaluation	15
Discussion	15
References	15
Appendix A.--Material balance for PRU test	17
Appendix B.--Process economics	20

ILLUSTRATIONS

1.	Location of McDermitt caldera	3
2.	Generalized process flowsheet	4
3.	Ball mill	5
4.	Drum pelletizer	5
5.	Direct-fired rotary roaster	6
6.	Leach tank	7
7.	PRU equipment	8
B-1.	Feed preparation section	24
B-2.	Roasting section	24
B-3.	Leaching section	25
B-4.	Evaporation section	25
B-5.	Lithium recovery section	26
B-6.	Crystallization section	26

TABLES

1.	Composition of McDermitt clay	4
2.	Effect of charge composition on lithium extraction under static conditions	10
3.	Effect of roasting temperature on lithium extraction under static conditions	10
4.	Effect of charge composition and roast temperature on lithium extraction under dynamic conditions	11
5.	Effect of solids content and wash water recycle on lithium extraction	11

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TABLES--Continued

		Page
6.	Effect of calcine particle size on lithium extraction	12
7.	Effect of leach time on lithium extraction	12
8.	Effect of calcine particle size and leach time on filtrate rates	12
9.	Results of product purification tests using simulated solutions	14
A-1.	Feed preparation	17
A-2.	Roast	17
A-3.	Leach	18
A-4.	Evaporation	18
A-5.	Product precipitation	19
A-6.	Crystallization	19
B-1.	Composition of McDermitt clay (dry basis)	20
B-2.	Estimated capital cost	21
B-3.	Estimated annual operating cost	23
B-4.	Raw material and utility requirements	23

UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	L/(m^2·h)	liter per square meter per hour
cm	centimeter	lb	pound
d/yr	day per year	M	thousand
ft	foot	m^3	cubic meter
g/L	gram per liter	mi	mile
gal	gallon	min	minute
h	hour	mL	milliliter
h/d	hour per day	MM	million
hp	horsepower	mm-diam	millimeter-diameter
in	inch	pct	percent
kg	kilogram	psi	pound per square inch
km	kilometer	ton/d	short ton per day
kW	kilowatt	W	watt
kW·h	kilowatt hour	wt pct	weight percent
L	liter	yr	year
L/kg	liter per kilogram		

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RECOVERY OF LITHIUM FROM A MONTMORILLONITE-TYPE CLAY

By R. H. Lien[1]

With an Appendix on Process Economics by D. A. Kramer

ABSTRACT

The Bureau of Mines investigated a roast-leach process for recovering a marketable lithium product from a montmorillonite-type clay deposit located on the Nevada-Oregon border. The clay sample treated in the investigation contained 0.6 wt pct Li.

The lithium recovery process consisted of several unit operations. The lithium silicate compounds in the clay were converted to Li_2SO_4 by roasting a pelletized mixture of clay, limestone, and gypsum at 900° C in a direct-gas-fired rotary roaster. Water-leaching the calcine at 40 pct solids extracted the Li_2SO_4. Lithium recovery from the leach solution involved concentrating the solution by evaporation, adding Na_2CO_3 to the concentrated solution to precipitate Li_2CO_3, and filtering the slurry to obtain the product. The product filtrate was recycled to the evaporator following a crystallization step. About 80 pct of the lithium in the clay was recovered as 99-pct-pure Li_2CO_3.

Process operating costs were estimated at \$2.12/lb Li_2CO_3 produced; the current Li_2CO_3 selling price is \$1.48/lb. Raw materials accounted for 30 pct of the total operating cost.

[1]Chemical engineer, Salt Lake City Research Center, Bureau of Mines, Salt Lake City, UT.

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INTRODUCTION

The Bureau of Mines investigated several processes for recovering lithium from a clay deposit located on the Nevada-Oregon border. Development of a process to recover lithium from this nonconventional, domestic resource would help meet the Bureau's goal of developing technology to help the Nation maintain an adequate minerals base for future economic and strategic needs.

The United States, the world's largest producer and consumer of lithium minerals and chemicals, is self-sufficient in lithium. Nearly all the Nation's lithium is recovered from spodumene deposits in North Carolina and subsurface brines in Nevada.

The largest end use for lithium is in aluminum potlines. In the aluminum cells, Li_2CO_3 is added to reduce electricity consumption and fluorine emissions. The ceramics, air conditioning, grease, synthetic rubber, and pharmaceutical industries also use lithium chemicals. Recently, lithium has gained importance in areas such as (1) low-density aluminum-lithium aircraft alloys, (2) lightweight batteries for use in electric automobiles and utility load-leveling purposes, and (3) nuclear fusion for use as a supply of tritium and as a coolant and heat transfer agent.

In the 1970's, several research and Government organizations discussed the possibility of domestic lithium shortages by the year 2000 (1-3).[2] These predictions were based primarily on rapid expansion of the lithium batteries market, principally in the area of electric automobiles. The development of thermonuclear energy was expected to have an impact on lithium reserves after the year 2000.[3]

Forecasts of lithium shortages prompted the U.S. Geological Survey (USGS), under a cooperative agreement with the Department of Energy, to search for alternative domestic lithium resources. The USGS identified a large deposit of lithium-bearing clay in the McDermitt caldera complex on the Nevada-Oregon border (fig. 1). The caldera complex, one of the largest in the world, comprises five overlapping and nested calderas (circular volcanic depressions). The principal clay deposit area measures 42 km long by 18 km wide.

The lithium-bearing clays are found primarily along the edge of the caldera in a crescent extending from the northeastern corner to the southwestern section. Lithium concentration in the clay is 0.1 to 0.36 pct in the northern deposits and 0.1 to 0.65 pct in the southern area.

These clay deposits can be considered a potential resource because of high lithium content in individual beds. Also, the beds have very little overburden. The amount of lithium in the caldera has been estimated as high as 10 MM tons.

The Bureau's Salt Lake City Research Center obtained about 6 M lb of clay from a discovery cut in the southwestern section of the caldera (fig. 1). A typical clay sample contained 0.6 wt pct lithium.

Bureau investigators studied several methods for extracting lithium from the McDermitt clay (5-9). The research included extensive laboratory studies on a lime-gypsum roast process that converts

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[2]Underlined numbers in parentheses refer to items in the list of references preceding the appendixes.

[3]Estimates of lithium demands have been revised because the lithium batteries market has not expanded as anticipated. Also, fusion power programs have encountered technological as well as funding problems, which has further clouded the lithium demand picture. In 1980, the Bureau of Mines predicted that development of lithium resources such as McDermitt clays would not be needed until at least the year 2050 (4). However, the recent development of aluminum-lithium aircraft alloys, which cut aircraft weight, resulting in considerable fuel savings, may cause a substantial increase in the demand for lithium.

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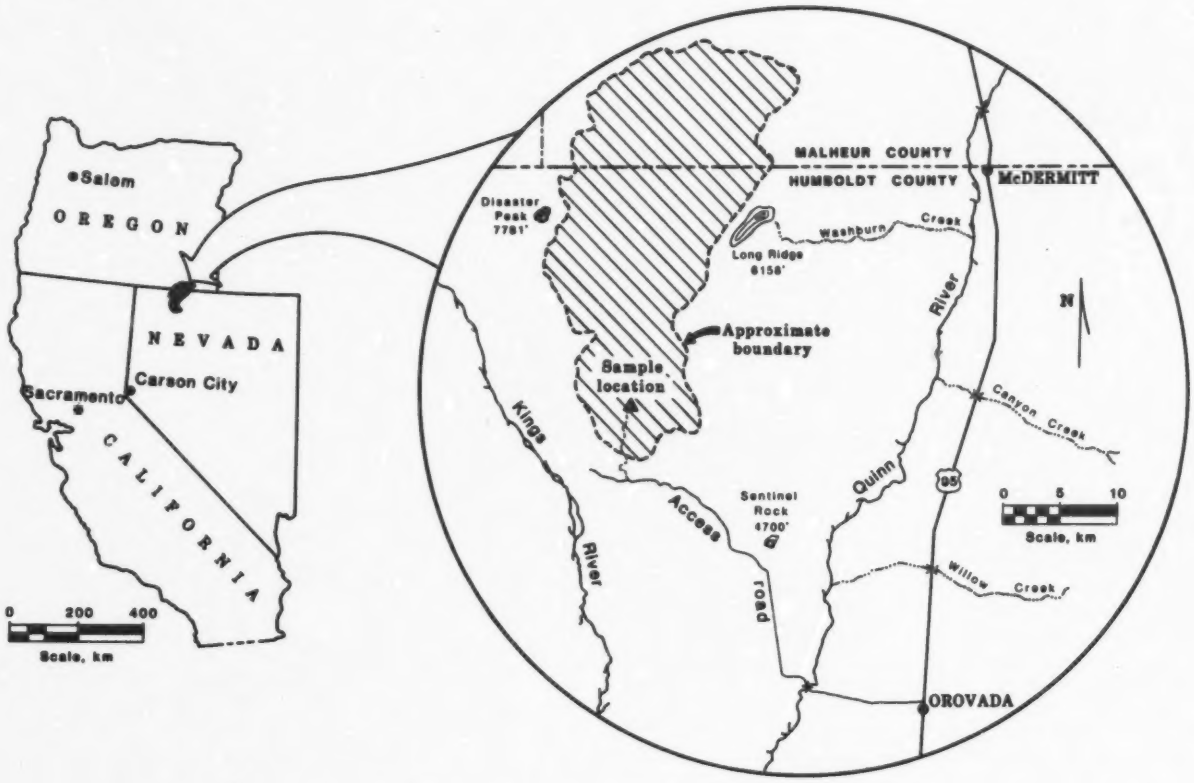

FIGURE 1. - Location of McDermitt caldera.

the lithium in the clay to a water-soluble product, without converting major contaminating elements such as aluminum and magnesium to water-soluble compounds (7). Water leaching extracts more than 80 pct of the lithium present in the ore, along with approximately equivalent percentages of the sodium and potassium. The leach solution is concentrated by evaporation, and a Li_2CO_3 product is precipitated by adding soda ash (Na_2CO_3). A process research unit (PRU) was built and operated to confirm the laboratory results and provide data for a cost evaluation.

This report presents the PRU operating procedures and conditions, test results, and material balances. A study or factored-type capital and operating cost evaluation is also included.

PROCESS DESCRIPTION

Figure 2 presents the generalized flowsheet for the PRU. The process involves several unit operations: feed preparation, roasting, leaching, evaporation, and crystallization.

A mixture of clay, limestone, and gypsum is pelletized, dried, and fed to the roasting furnace. Roasting the pellets at 900° C converts the lithium in the clay to water-soluble Li_2SO_4. Water-leaching the calcine produces a solution containing lithium, potassium, sodium, and a small amount of calcium, all as sulfates. The leach solution, together with recycled product wash and mother liquor, is concentrated by evaporation. During this concentration operation, carbonate ion present in the recycle solutions causes calcium to precipitate as $CaCO_3$, which is removed by filtration. The concentrated solution is then heated to boiling, and Na_2CO_3 is added to precipitate Li_2CO_3 product.

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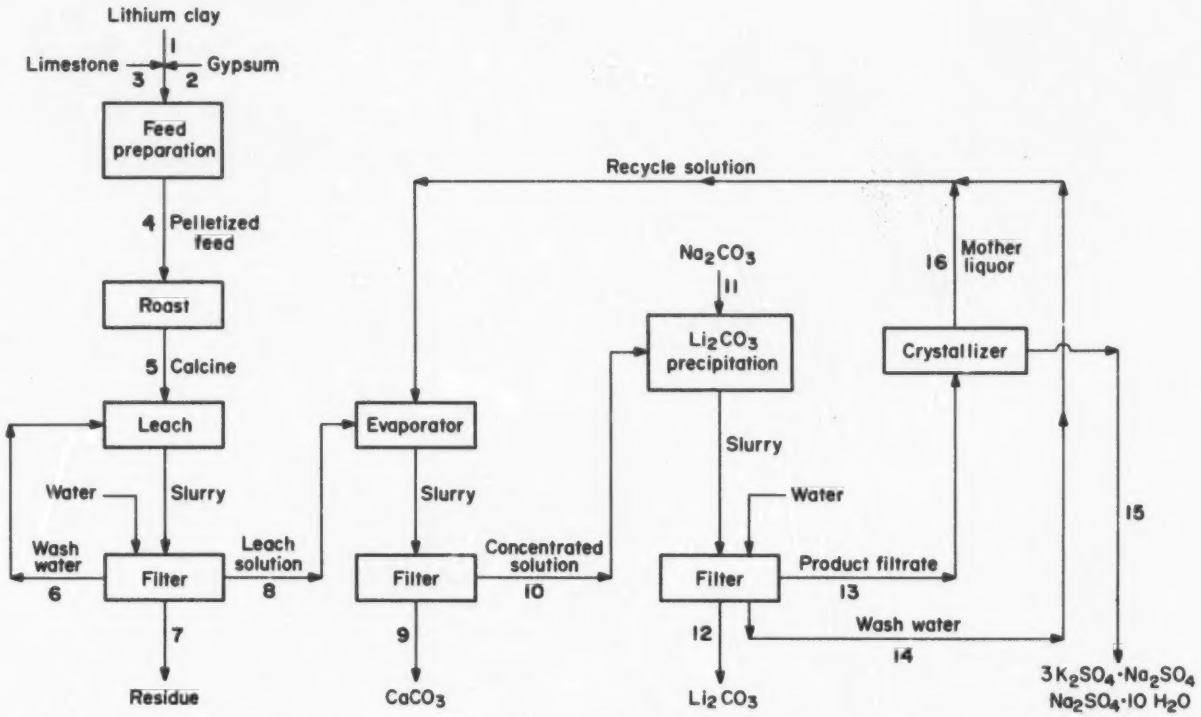

FIGURE 2. - Generalized process flowsheet. Material balances for numbered streams are presented in appendix A tables.

The product filtrate contains about 3 g/L Li. To recover this lithium, the solution is cooled to crystallize $3K_2SO_4 \cdot Na_2SO_4$ and $Na_2SO_4 \cdot 10H_2O$, and then recycled to the evaporation step.

Removing calcium from the leach solution as $CaCO_3$ prevents calcium contamination of the product. The solution recycled to the evaporation step contains sufficient carbonate ion, as either Li_2CO_3 or excess Na_2CO_3, to precipitate over 99 pct of the calcium contained in the leach solution.

MATERIALS

Table 1 shows the composition of a typical clay sample. X-ray diffraction studies, together with the clay's chemical properties, indicated that the clay is chiefly montmorillonite. The lithium occurs in the clay mineral hectorite $[Na_{0.33}(Mg, Li)_3Si_4O_{10}(F, OH)_2]$ and an unnamed clay mineral similar to hectorite but containing more aluminum (10).

TABLE 1. - Composition of McDermitt clay, percent

Al.....	3.1	Li.....	0.6
Ca.....	1.8	Mg.....	9
F.....	2	Na.....	.58
Fe.....	2.5	Total SiO_2..	53
K.....	3.7	Free SiO_2...	15

Mineralogical studies showed that the lithium is evenly disseminated throughout the clay; thus, physical beneficiation techniques would not upgrade the lithium content.

The feed for the roast was a mixture of clay, limestone, and agricultural-grade gypsum. The Li_2CO_3 was precipitated from the concentrated solution by adding dense soda ash (Na_2CO_3).

Simulated solutions used in laboratory evaporation and product purification tests were made up with reagent-grade chemicals.

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PRU EQUIPMENT

The feed for the roast was ground and mixed in the ball mill (40-cm-diam by 60-cm-length) shown in figure 3. The minus 100-mesh mixture was pelletized with water in the drum pelletizer (50-cm-diam) shown in figure 4.

The pellets were roasted in a direct-fired rotary roaster. The roaster, shown in figure 5, was fired with natural gas and had a working capacity of 0.014 m^3. The roast product (calcine) was leached with water in the 208-L baffled, polyethylene vessel shown in figure 6. A propeller mixer, driven by a 0.75-hp direct-drive motor, agitated the slurry.

Figure 7 shows the evaporation, filtration, and product precipitation equipment. To recover the leach solution, the slurry from the leach tank was transferred by gravity flow to a pan filter with a surface area of 0.8 m^2. The filter cloth was a medium-weight cotton twill. A tubing pump transferred the leach solution to a 208-L stainless steel tank for concentration. The tank was equipped with a 9,000-W over-the-side immersion heater. The concentrated solution was filtered on a tabletop Buchner funnel to remove $CaCO_3$. A tubing pump transferred the concentrated solution to the product precipitation unit, which consisted of a 24-L stainless steel vessel equipped with a 1,000-W over-the-side immersion heater. An impeller mixer, driven by a 0.25-hp direct-drive motor, agitated the solution. The Li_2CO_3 product was recovered and washed on a Buchner funnel.

The product filtrate was cooled by refrigeration to crystallize $Na_2SO_4 \cdot 10H_2O$ and $3K_2SO_4 \cdot Na_2SO_4$.

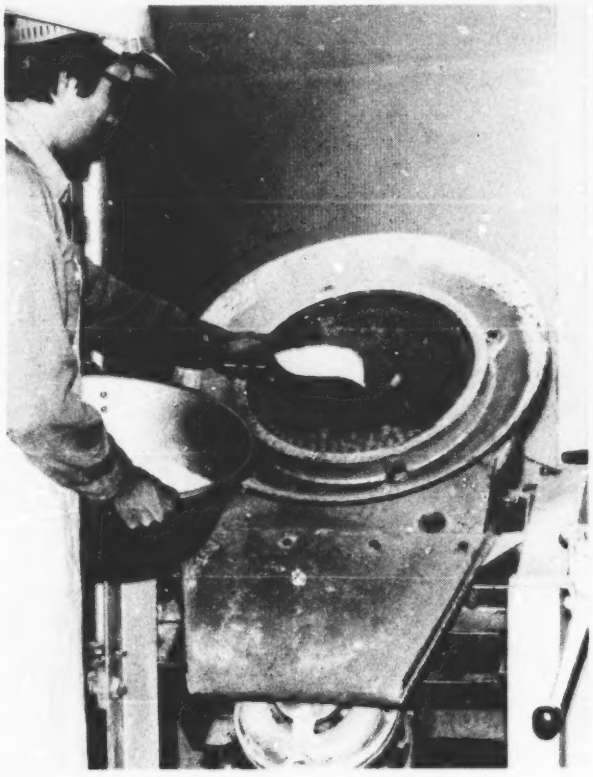

FIGURE 3. - Ball mill.

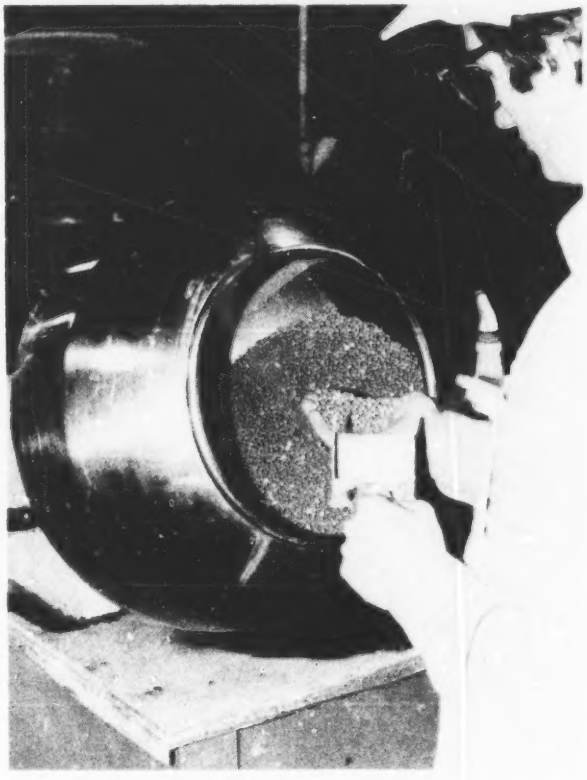

FIGURE 4. - Drum pelletizer.

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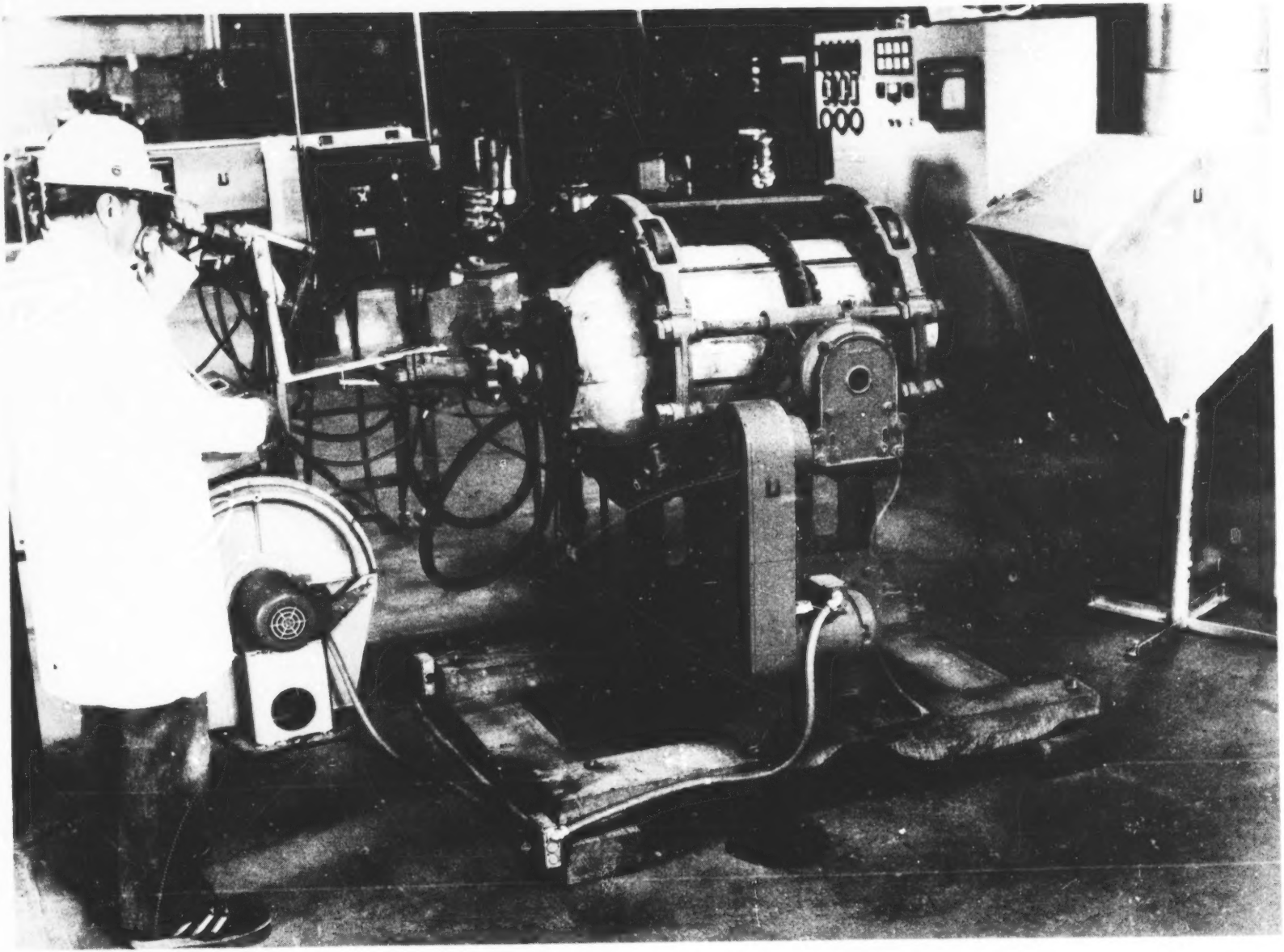

FIGURE 5. - Direct-fired rotary roaster.

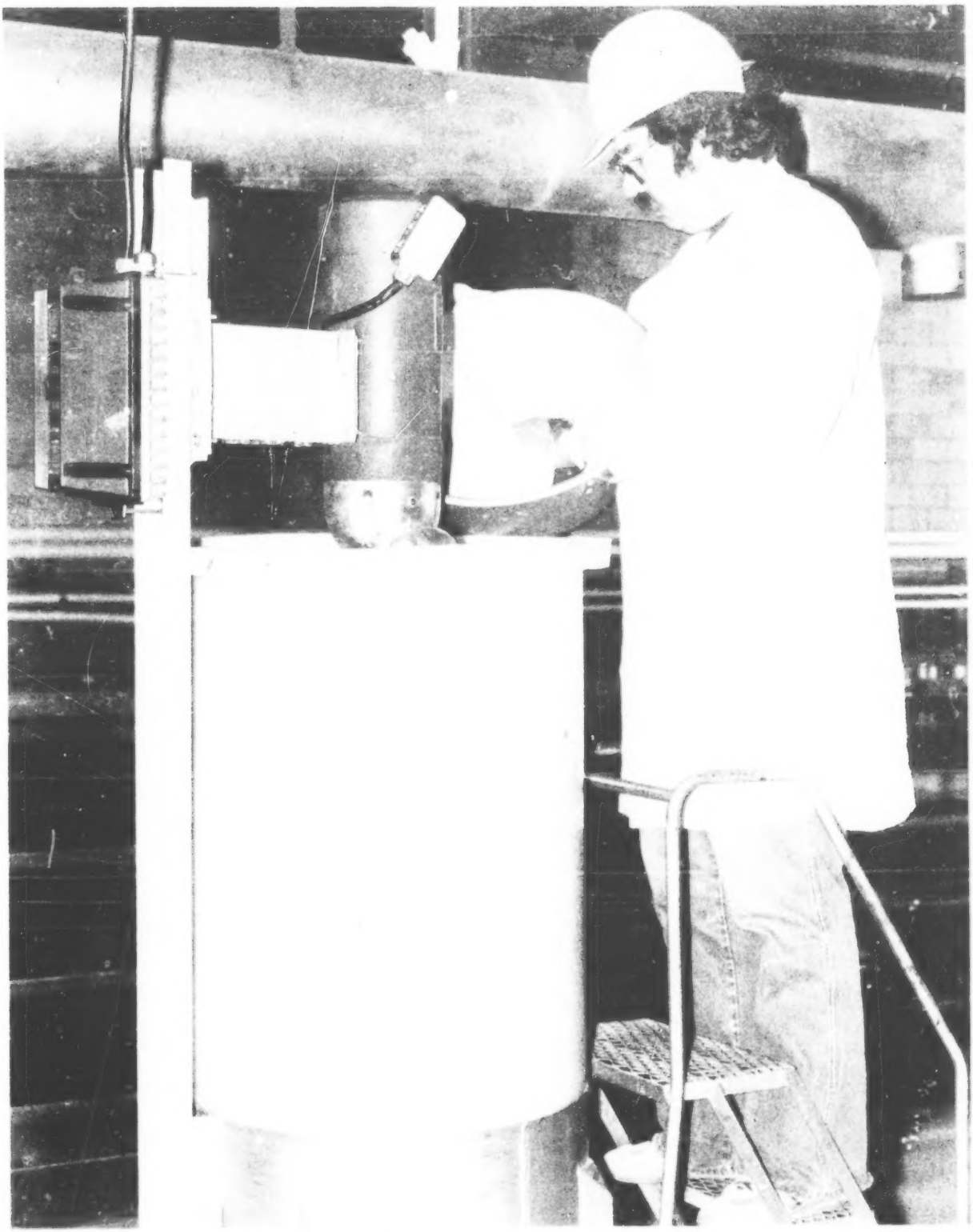

FIGURE 6. - Leach tank.

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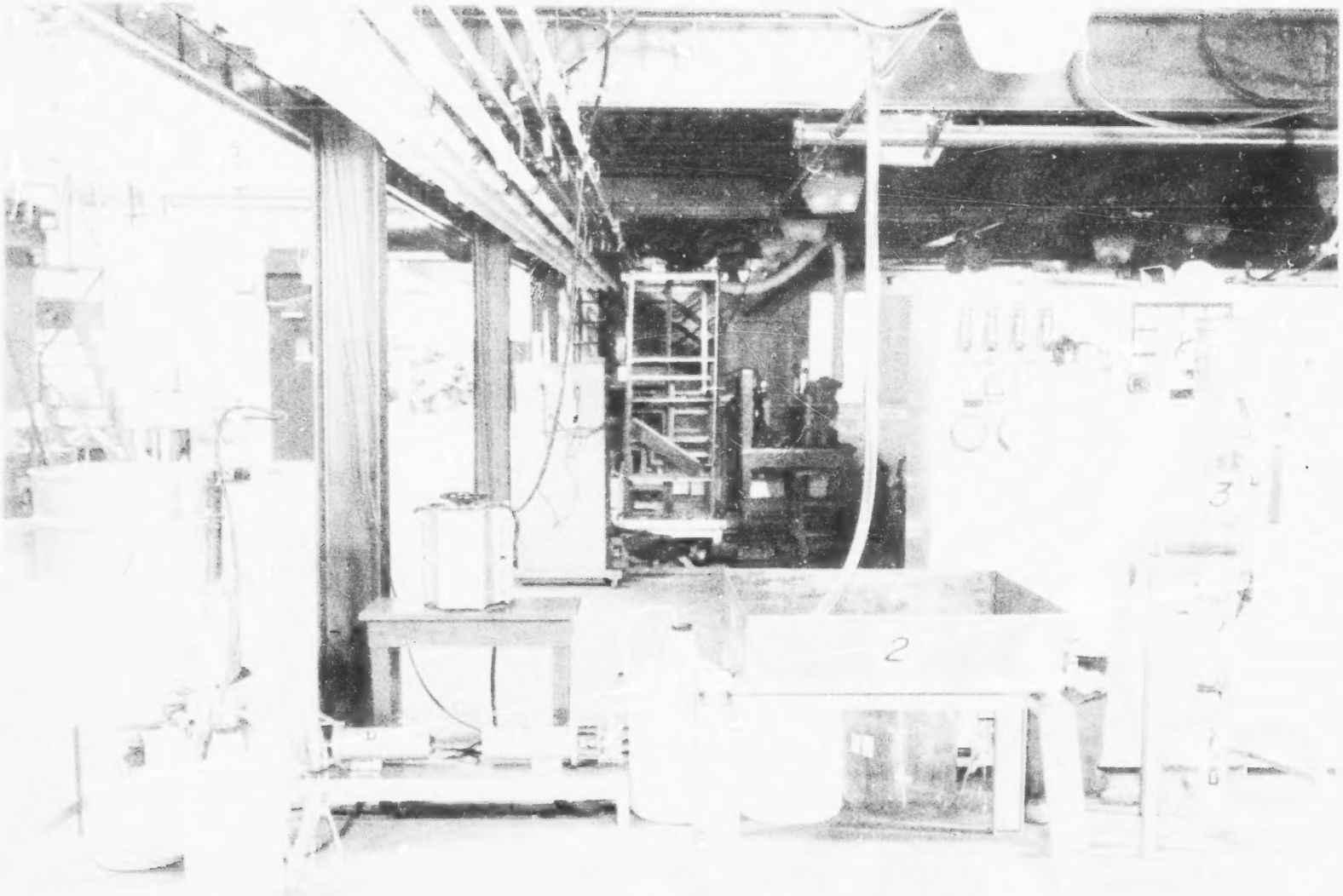

FIGURE 7. - PRU equipment. , Evaporator, , pan filter, , product precipitation unit.

OPERATING CONDITIONS AND TEST RESULTS

Process unit operations were studied in a PRU and in laboratory tests to determine operating conditions that would (1) maximize lithium recovery, (2) minimize process operating costs, and (3) produce a high-purity product.

Appendix A presents a material balance for a typical PRU test using a 5:3:3 ratio of clay, limestone, and gypsum; a 5:2:2 ratio might also be considered since reagent and utility costs would be lower.

FEED PREPARATION

The McDermitt clay contains lithium principally as hectorite. To convert the lithium to Li_2SO_4, the clay was mixed with limestone and gypsum and roasted.

Feed preparation entailed grinding and mixing the ingredients (clay, limestone, and gypsum) for 1 h in a ball mill. The resultant mixture (80 pct finer than 200 mesh) was pelletized with water to produce nominal 6.5-mm-diam pellets. These pellets contained up to 20 pct moisture and were dried at 70° C before roasting.

ROASTING

Roasting studies consisted of tube furnace tests, batch tests conducted in an electrically heated muffle furnace, and continuous tests made in a gas-fired rotary roaster. The tests were conducted to determine the reaction mechanism and to establish optimum roasting conditions (7).

Roasting Mechanism

The pelletized mixture of clay, limestone, and gypsum was roasted to convert lithium in the clay to water-soluble Li_2SO_4. Data obtained from exploratory tube furnace tests (7) indicated that Li_2SO_4 was formed by the following reactions (for simplicity, lithium-silicate minerals such as hectorite are represented by $Li_2Si_2O_5$):

$$CaSO_4 \cdot 2H_2O + SiO_2 \longrightarrow CaSiO_3 + SO_2 + 1/2O_2 + 2H_2O \quad (1)$$

$$Li_2Si_2O_5 + SO_2 + 1/2O_2 \longrightarrow Li_2SO_4 + 2SiO_2 \quad (2)$$

Limestone limited the back reaction of free SiO_2 with Li_2SO_4. Free silica appeared to react with CaO to form a calcium silicate.

Batch Tests

Extensive batch testing was conducted in an electrically heated muffle furnace to determine the effects of charge composition, roasting time, and temperature on lithium extraction (7-8). Specifically, the following conditions were investigated: (1) preroast mixtures (clay-limestone-gypsum) ranging from 5:0:6 to 5:6:0, (2) roasting times of 1 to 4 h, and (3) roasting temperatures of 750° to 1,050° C.

Reagent-grade $CaCO_3$ and $CaSO_4 \cdot 2H_2O$ were used in these tests. The ingredients were pelletized and roasted. The resultant calcines were water-leached to determine lithium extraction.

Data presented in table 2 show the effect of charge composition on lithium extraction. These tests were conducted at 1,000° C for 1 h. Of the compositions tested, a 5:3:3 mix was optimum with a lithium extraction of 88.4 pct. Lithium extraction from clay roasted alone (5:0:0) was only 0.1 pct.

Table 3 shows the effect of roasting temperature on lithium extraction. In these tests, a 5:3:3 mixture was calcined for 1 h at 750° to 1,100° C. The optimum extraction was attained at 1,000° C. Roasting for periods longer than 1 h did not improve lithium extraction. Prolonged roasting for 4 h at elevated temperatures (above 850° C) slightly reduced lithium recovery.

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TABLE 2. - Effect of charge composition on lithium extraction under static conditions[1]

Weight ratio			Li extraction,
Clay	$CaCO_3$	$CaSO_4 \cdot 2H_2O$	pct
5	0	6	61.6
5	1	5	70
5	2	3	83.7
5	2	4	85.8
5	3	2	87.6
5	3	3	88.4
5	3	4	87.8
5	4	3	80.9
5	4	4	78.9
5	5	3	81.2
5	5	1	73.3
5	6	0	12.5
5	0	0	.1

[1]Each mixture was roasted at 1,000° C for 1 h.

TABLE 3. - Effect of roasting temperature on lithium extraction under static conditions[1]

	Li extraction, pct
750° C.....	59.4
800° C.....	73.3
850° C.....	79.5
900° C.....	83.8
950° C.....	85.6
1,000° C.....	88.4
1,050° C.....	83
1,100° C.....	65.7

[1]Roasting time was 1 h.

Tests were also conducted to study the effect of naturally occurring limestone and gypsum on lithium recovery (7). In these tests, the quantities of limestone and gypsum mixed with the clay were calculated to produce an equivalent 5:3:3 mix. The equivalence was based on the CaO content of reagent-grade $CaCO_3$ and $CaSO_4 \cdot 2H_2O$. The mixtures were ground to minus 200 mesh, pelletized, and roasted at 1,000° C for 1 h. Lithium extractions ranged from 83 to 85.5 pct, compared with 88.4 pct for roasts using reagent-grade chemicals. The decrease in extraction was attributed to the coarser particle size of the natural materials.

Continuous Roast Tests

Larger scale testing was conducted in a gas-fired rotary roaster to study the dynamic variables affecting the calcine and to confirm laboratory test results.

Initially, a series of batch tests was conducted in the rotary roaster to determine optimum roast temperature and retention time (7). In these tests, small charges (500 g) of pelletized 5:3:3 mix were roasted. The test results showed 900° C and a 2-h retention time to be optimum. The initial continuous roasting tests were conducted at these conditions.

The primary objective of the continuous testing was to generate calcine for use in PRU leach tests. An equivalent 5:3:3 mixture of clay, limestone, and gypsum was used because batch testing established this mix as optimum. The pelletized feed was charged to the roaster in 600-g increments every 5 min. Generally, each test produced 36 kg of calcine in about 6-1/2 h operating time.

The final phase of the continuous roast work involved investigating the effects of charge composition and roast temperature on lithium extraction. A series of tests was conducted in which various mixes were roasted. Lithium extraction was determined by water-leaching composite samples of the calcines.

Test results, presented in table 4, show that lithium extractions of at least 80 pct were attained with a wide range of clay-limestone-gypsum ratios. Also, the data indicate that good lithium extraction was achieved over a temperature range of 850° to 975° C. The 5:3:3 mix was chosen as the basis for a cost evaluation because other mixtures, such as 5:2:2, had not been adequately studied at the time the evaluation was begun. Details are presented in appendix B.

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TABLE 4. - Effect of charge composition and roast temperature on lithium extraction under dynamic conditions

Weight ratio			Roast temp, °C[1]	Roasts at given conditions	Li extraction, pct[2]
Clay	$CaCO_3$	$CaSO_4 \cdot 2H_2O$			
5	1	1	900	1	59
5	1.5	1.5	900	1	80.5
5	1.5	1.5	975	1	81
5	2	1	900	1	81.6
5	2	2	850	1	84.6
5	2	2	900	3	84.3-86.9
5	2	2	950	1	89.3
5	2	3	900	1	84.4
5	3	2	900	2	86.8-88.5
5	3	3	900	17	83.3-86.6
5	3	4	900	1	85.1

[1]Retention time was 2 h.

[2]For analysis, calcine samples were leached with water at 25 pct solids; the residues were then washed. Lithium extraction was based on calcine and residue analyses.

LEACHING

The objective of the PRU leach tests was to determine the relationship between leach-system variables and optimal lithium extraction. The following variables were studied: (1) leach pulp percent solids, (2) wash water recycle, (3) calcine particle size, and (4) leach time.

The calcines leached in these tests were generated by roasting 5:3:3 mixtures of clay, limestone, and gypsum. Generally, 32 kg of calcine was water-leached in each test. The slurry was filtered to recover the leach solution. The filter cake was then washed and discarded.

Solids Content and Wash Water Recycle

Studies were conducted to minimize the volume of leach solution produced and thereby reduce subsequent evaporation requirements. The volume of solution can be reduced by increasing the percent solids in the leach slurry and recycling the calcine wash water.

A series of 30-min leach tests was conducted to study the effect of percent solids and wash water recycle on lithium extraction. The test results, presented in table 5, show that the calcine was leached effectively at 40 pct solids with recycled wash water. At 50 pct solids, the lithium extraction decreased. Since the wash water was recycled to the leach step, the volume of wash water used was equal to the volume of water required for the next leach.

TABLE 5. - Effect of solids content and wash water recycle on lithium extraction, percent[1]

Solids content, pct	Leached with--	
	Fresh water	Recycled wash water
25[2].....	83.4	82.7
40[3].....	81.4	83.6
50[3].....	80.9	69.4

[1]Extractions based on lithium concentrations in calcine and leached residue.

[2]96-L leach and wash water volume.

[3]48-L leach and wash water volume.

Calcine Particle Size and Leach Time

The calcine pellets do not break apart during the leach. If a coarse particle could be leached effectively, grinding requirements would be minimized. Therefore, a series of tests was conducted to study the effect of calcine particle size on lithium extraction.

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The calcine was leached for 30 min at 40 pct solids using recycled wash water. The test results, given in table 6, show that the 30-min leach extracted the lithium equally well from all particle sizes tested.

TABLE 6. - Effect of calcine particle size on lithium extraction

Particle size	Li extraction, pct[2]
Minus 100 mesh.....	82.6
Coarse-crushed pellets[1]..	83.6
Whole pellets.....	83.9

[1]Screen analysis of calcine particles was 70 pct plus 10 mesh.

[2]Extractions based on lithium concentrations in calcine and leached residue.

To determine the effect of leach time on lithium extraction, a series of tests was conducted with coarse-crushed and whole pellets. The pellets were leached at 40 pct solids in recycled wash water. The test results, presented in table 7, show that no more than a 5-min leach was required to extract the lithium from coarse-crushed pellets. However, whole pellets were not effectively leached in 5 min.

TABLE 7. - Effect of leach time on lithium extraction

Particle size	Leach time, min	Li extraction, pct
Coarse-crushed pellets.....	5	84.2
	15	84.2
	30	83.6
	60	83.1
Whole pellets.....	5	70.7
	30	83.9
	60	84.2

Although the pellets did not break apart during the leach, prolonged agitation generated fines which affected filtration rates. The data presented in table 8 show that filtrate rates decreased with increased leach time. Also, the rates decreased as calcine particle size increased. For 30-min leaches, the whole pellet slurry filtered slowly because the filter cloth was blinded with fines. As calcine particle size decreased, the fines tended to remain on top of the filter cake, allowing faster filtration.

TABLE 8. - Effect of calcine particle size and leach time on filtrate rates

Calcine particle size	Leach time, min	Filtrate rate,[1] $L/(m^2 \cdot h)$
Minus 100 mesh...	30	86
Coarse-crushed pellets.....	5	155
	15	111
	30	68
	60	67
Whole pellets....	5	150
	30	42

[1]Rates calculated from data obtained by filtering leach slurry on process pan filter.

The test results indicate that the calcine particle size used in the leach would be determined by evaluating the operating costs, including grinding, filtration, and agitation costs.

Summary of Leach Test Results

The PRU results show a maximum lithium extraction from the calcine of 82 to 84 pct. Based on test data, the preferred extraction procedure involved leaching coarse-crushed pellets with recycled wash water for 5 min at 40 pct solids. Leaching at ambient temperature produced a solution containing 2.5 to 3.0 g/L Li.

EVAPORATION

As figure 2 shows, the evaporator was fed with leach solution and solution recycled from the previous test. The recycled solution (mother liquor plus product wash) accounted for about 20 pct of the total volume in the evaporator.

In addition to concentrating the solution, calcium was removed from the leach

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solution in this step of the process. The leach solution was saturated with $CaSO_4$ (about 0.6 g/L Ca^{2+}). Extensive laboratory testing showed that reducing the calcium ion concentration to about 0.015 g/L prevented calcium contamination of the product.

The evaporation procedure involved the following steps:

1. The solution (leach plus recycle) was evaporated to about 50 pct of its original volume and then filtered to remove $CaCO_3$. Carbonate ion (approximately 15 g/L) present in the recycled solution precipitated over 99 pct of the calcium contained in the leach solution.
2. The filtrate was returned to the evaporator. Evaporation continued until the solution was reduced to 20 pct of its original volume.
3. The hot concentrated solution, containing 12 to 13 g/L Li, was transferred to the product precipitation step. Generally this concentrated solution was cloudy because a small amount of Li_2CO_3 precipitated during evaporation.

PRODUCT PRECIPITATION

Lithium recovery involved heating the concentrated solution to boiling and adding a stoichiometric amount of Na_2CO_3 to precipitate a Li_2CO_3 product. The objective of this step was to recover a product of at least 99-pct purity.

Initially, the product was recovered from the hot solution by vacuum filtration and then dried. This procedure yielded a product of about 80-pct purity with the principal contaminants being Na_2SO_4 and K_2SO_4. Numerous tests were conducted in the PRU and laboratory (using PRU leach solution) to investigate product purification techniques. Test results were erratic because precise control of solution concentration was difficult; therefore, simulated solutions were used to study operating variables.

A series of laboratory tests was conducted using 1-L batches of simulated concentrated solution (made up with reagent chemicals) containing 97 g/L Li_2SO_4, 158 g/L K_2SO_4, and 87 g/L Na_2SO_4. Adding a stoichiometric amount of Na_2CO_3 to the hot solution precipitated Li_2CO_3. Product filtration and washing procedures were then studied. From the test results presented in table 9, the following observations were made:

1. Pressure filtration yielded a product of higher purity than vacuum filtration by reducing the moisture content of the filter cake.
2. On the pressure filter, 4 to 6 L of wash water per kilogram of dry product was required to produce a 99-pct-pure product. A much higher volume of water was needed to produce a comparable product by vacuum filtration.
3. For pressure filtration, wash water volumes above 6 L per kilogram of dry product did not further improve product purity. Also, single-stage washing was as effective as either multistage washing or product reslurry.
4. Adding Na_2CO_3 as a saturated solution rather than as a dry powder had little effect on product purity. However, this procedure generated a coarse grainy product in contrast to the fine powdery product obtained by adding dry Na_2CO_3.

The wash water and product filtrate recovered in these tests contained 14 to 16 g/L Li_2CO_3. The wash was recycled to the evaporator. After a crystallization step, the product filtrate was also recycled.

CRYSTALLIZATION

In addition to residual Li_2CO_3, the product filtrate contained high concentrations of K_2SO_4 and Na_2SO_4 (over 150 g/L of each). If the solution is to be recycled, the buildup of these salts must be prevented.

Laboratory and PRU tests showed that the most effective method for reducing the sulfate concentration involved crystallizing the salts by chilling the solution to between 0° and -4° C (below -4° C, the solution freezes). The mother liquor, which contained 70 g/L Na_2SO_4 and 100 g/L K_2SO_4, was recovered by either vacuum or pressure filtration. Pressure filtration tended to reduce lithium loss by decreasing the amount of mother liquor present in the filter cake.

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TABLE 9. - Results of product purification tests using simulated solutions

Wash water volume, mL	Water per dry product, L/kg	Product analysis, wt pct[1]		Comments[2]
		K	Na	
Vacuum filtration:[3]				
0.....	0	4.1	3.9	No wash.
300.....	5.7	.86	.6	Single wash.
600.....	12.8	.4	.3	Do.
700.....	15.3	.12	.14	Do.
800.....	17.5	.16	.16	Do.
Pressure filtration:[4]				
0.....	0	2.3	2	No wash.
	0	1.9	1.8	Na_2CO_3 added as saturated solution; no wash.
	0	1.6	1.5	Do.
100.....	2	.3	.3	Single wash.
200.....	4.1	.17	.17	Do.
300.....	6.1	.16	.15	Do.
	6.3	.16	.1	Product reslurried with 200 mL, filtered, then washed with 100 mL.
	6.6	.26	.18	Na_2CO_3 added as saturated solution; single wash.
	6.2	.16	.16	3 separate 100-mL washes.
600.....	12.9	.18	.16	Single wash.
	13.2	.14	.14	6 separate 100-mL washes.
800.....	17.7	.18	.2	Single wash.

[1]K and Na present as sulfates; product also contained traces of Ca (0.02 wt pct) and Mg (0.01 wt pct), probably present as carbonates.

[2]Except as indicated, Na_2CO_3 was added to the concentrated solution as dry powder.

[3]Carried out on a laboratory-size Buchner funnel.

[4]Conducted with a tabletop filter connected to a 45-psi air supply.

The filter cake was a mixture of glauber salts ($Na_2SO_4 \cdot 10H_2O$) and glaserite ($3K_2SO_4 \cdot Na_2SO_4$).

Laboratory tests showed that, if desired, glaserite and glauber salts could be recovered separately by a two-step crystallization procedure. At solution temperatures down to about 17° C, glaserite crystallized. The salt was recovered by vacuum filtration and analyzed as 33 wt pct K, 8 wt pct Na, and <0.1 wt pct Li. Further cooling of the solution (to as low as -4° C) crystallized glauber salts. These salts were recovered by pressure filtration and dried. The dried salts contained 28 wt pct Na, 6 wt pct K (a small amount of glaserite crystallized with the $Na_2SO_4 \cdot 10H_2O$), and 0.15 wt pct Li.

OVERALL LITHIUM RECOVERY

PRU roast-leach test results (tables 5-7) indicate 82- to 84-pct Li extraction as optimum. Treating the leach solution by the methods specified resulted in 95- to 98-pct recovery of the contained lithium. Losses occurred in $CaCO_3$ filtration (0.5-pct loss) and in the crystallization step (2- to 5-pct loss depending on the filtration method used to separate the mother liquor from the salts). Overall, 78 to 82 pct of the lithium contained in the clay was recovered as 99-pct-pure Li_2CO_3.

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MATERIAL BALANCE AND ECONOMIC EVALUATION

Appendix A shows a material balance for a typical PRU test using a 5:3:3 mixture of clay, limestone, and gypsum. The Bureau's Process Evaluation Office at Avondale, MD, prepared a cost evaluation based on the PRU material balance, filtration rate data, flowsheet, and process description supplied by the Salt Lake City Research Center.

The process evaluation, presented in appendix B, estimates the operating cost of the process at \$2.12/lb Li_2CO_3 produced. (The current selling price of Li_2CO_3 is \$1.48/lb). The evaluation identifies the high-cost areas of the process as reagents, utilities, and depreciation. A summary of capital and operating costs for a plant processing 1,000 ton/d of McDermitt clay is presented in tables B-2 and B-3.

Table B-3 shows raw materials and utilities to be the high-cost areas of the process. Reagent costs are high because of the plant location, which necessitates shipping large quantities of limestone and gypsum long distances. Reagent costs could be reduced if (1) sources of limestone and gypsum in close proximity to the plant were identified and developed and (2) the quantity of reagents used in the roast feed was reduced. (Table 4 indicates that a 5:2:2 ratio of clay, limestone, and gypsum is as effective as a 5:3:3 mix.) In addition, high fuel costs associated with roasting would be reduced by decreasing the quantity of limestone and gypsum in the roast feed.

DISCUSSION

PRU studies demonstrated a process for recovering lithium from the lithium-enriched clays of the McDermitt caldera. The process involves the following operations: (1) roasting a mixture of clay, limestone, and gypsum, (2) leaching the calcine with water, (3) concentrating the leach solution by evaporation, and (4) adding Na_2CO_3 to the concentrated solution to precipitate Li_2CO_3. About 80 pct of the lithium in the clay was recovered as 99-pct-pure Li_2CO_3.

Based on the \$1.48/lb Li_2CO_3 selling price, the process is not economical because of high reagent, utility, and depreciation costs. Reagent and utility costs associated with feed preparation and roasting can be reduced by using a 5:2:2 roast mix rather than 5:3:3; also, a reduction in reagent shipping charges, through reduced reagent requirements and/or development of closer reagent sources, would significantly decrease process operating costs. However, these savings would not make the process economic.

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APPENDIX A.--MATERIAL BALANCE FOR PRU TEST[1]

TABLE A-1. - Feed preparation

Stream	Li	K	Na	Ca	Mg	SO_4	CO_3	H_2O[2]	Other	Total
Wt, g:										
1.....	108.1	647.3	115.9	327.8	1,645.15	12.75	254.9	842.5	14,255.6	18,210
2.....	0	8.63	4.32	2,568.02	10.79	5,729.49	0	2,148.29	320.46	10,790
3.....	0	11.46	6.88	4,389.2	150.13	3.44	6,898.9	0	0	11,460
4[3].....	108.1	667.39	127.1	7,285.02	1,806.07	5,745.68	7,153.8	2,990.79	14,576.06	40,460
wt pct:										
1.....	0.59	3.55	0.64	1.8	9	0.07	1.4	4.6	78.28	100
2.....	0	.08	.04	23.8	.1	53.1	0	19.91	2.97	100
3.....	0	.1	.06	38.3	1.31	.03	60.2	0	0	100
4[4].....	.27	1.65	.31	18.01	4.46	14.2	17.68	7.39	36.03	100

[1]Streams are identified in figure 2.

[2]The clay-limestone-gypsum mixture was pelletized with 6.587 L water. The pellets were dried at 70° C to drive off this water.

[3]The weight of stream 4 represents the combined weights of streams 1-3.

[4]The composition of stream 4 represents the overall composition of streams 1-3.

TABLE A-2. - Roast

Stream	Li	K	Na	Ca	Mg	SO_4	CO_3	F	H_2O	Other	Total[1]
Wt, g:											
4.....	108.1	667.39	127.1	7,285.02	1,806.07	5,745.68	7,153.8	509.8	2,990.79	14,066.26	40,460
5.....	108.1	667.39	127.1	7,285.02	1,806.07	5,493.93	0	276.5	0	16,015.89	31,780
wt pct:											
4.....	.27	1.65	.31	18.01	4.46	14.2	17.68	1.26	7.39	34.77	100
5.....	.34	2.1	.4	22.92	5.68	17.29	0	.87	0	50.4	100

[1]The difference in total weight between the streams was accounted for in the roaster offgas. Excluding combustion gases, the offgas contained 167.83 g SO_2, 41.96 g O_2, 5,246.12 g CO_2, 2,990.79 g H_2O, and 233.3 g F.

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17

TABLE A-3. - Leach

Stream	Li	K	Na	Ca	Mg	SO_4	H_2O[1]	Other	Total
Wt, g:									
5	108.1	667.39	127.1	7,285.02	1,806.02	5,493.93	0	16,292.39	31,780
6	44.2	293	49	24.5	1.44	830.45	48,000	0	49,243
7	17.7	238.4	28.1	7,265.2	1,805.08	4,087.96	15,395	16,292.39	45,129.83
8	90.4	428.99	99	19.82	.99	1,405.97	32,605	0	34,650.17
wt pct:									
5	0.34	2.1	0.4	22.92	5.68	17.29	0	51.27	100
7	.04	.53	.06	16.1	4	9.06	34.11	36.10	100
g/L:									
6	0.92	6.1	1.02	0.51	0.03	17.29	NAp	0	NAp[2]
8	2.74	13	3	.6	.03	42.61	NAp	0	NAp[3]

NAp Not applicable.
[1]The filter cake was washed with 48 L water.
[2]The total volume of stream 6 was 48.04 L solution with a specific gravity of 1.025.
[3]The total volume of stream 8 was 33 L solution with a specific gravity of 1.05.

TABLE A-4. - Evaporation

Stream	Li	K	Na	Ca	Mg	SO_4	CO_3	H_2O[1]	Total
Wt, g:									
8	90.4	428.99	99	19.32	0.99	1,405.97	0.0	32,605	34,650.17
9	.45	1	.5	19.79	.99	2.27	34.06	25.6	84.66
10	119.3	681.97	237.86	.098	ND	2,007.13	91.38	7,813.98	10,952.17
14	7.09	10.57	10.57	.014	ND	35.07	30.41	2,798.28	2,892
16	22.26	243.41	128.79	.054	ND	568.36	95.48	5,217.7	6,276
wt pct:									
9	.53	1.18	.59	23.38	1.17	2.68	40.23	30.24	100
g/L:									
8	2.74	13	3	.6	.03	42.61	0	NAp	NAp[2]
10	13.42	76.71	26.26	.01	<.01	225.77	10.33	NAp	Nap[3]
14	2.5	3.73	3.73	.005	<.01	12.37	10.7	NAp	NAp[4]
16	4.11	44.99	23.81	.01	<0.1	105.06	17.65	NAp	NAp[5]

NAp Not applicable. ND Not determined.
[1]Solution was concentrated by evaporating approximately 32.8 L water.
[2]The total volume of stream 8 was 33 L solution with a specific gravity of 1.05.
[3]The total volume of stream 10 was 8.89 L solution with a specific gravity of 1.232.
[4]The total volume of stream 14 was 2.835 L solution with a specific gravity of 1.02.
[5]The total volume of stream 16 was 5.41 L solution with a specific gravity of 1.16.

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TABLE A-5. - Product precipitation

Stream	Li	K	Na	Ca	SO_4	CO_3	H_2O[1]	Total
Wt, g:								
10.....	119.39	681.97	237.86	0.098	2,007.13	91.83	7,813.98	10,952.17
11.....	0	0	321.7	.146	0	419.83	0	741.68
12.....	88.59	.76	.76	.09	2.52	379.81	104.43	576.96
13.....	23.62	670.64	548.23	.14	1,969.54	101.44	7,709.55	11,023.2
14.....	7.09	10.57	10.57	.014	35.07	30.41	2,798.28	2,892
wt pct:								
11.....	.0	.0	43.4	.02	.0	56.6	.0	100
12.....	15.35	.13	.13	.016	.44	65.83	18.1	100
g/L:								
10.....	13.42	76.71	26.76	.01	225.77	10.33	NAp	Nap[2]
13.....	2.74	77.8	63.6	.016	228.48	11.76	NAp	Nap[3]
14.....	2.5	3.73	3.73	.005	12.37	10.7	NAp	NAp[4]

NAp Not applicable.
[1]Product was washed with approximately 2.8 L hot water.
[2]The total volume of stream 10 was 8.89 L solution with a specific gravity of 1.232.
[3]The total volume of stream 13 was 8.62 L solution with a specific gravity of 1.279.
[4]The total volume of stream 14 was 2.835 L solution with a specific gravity of 1.02.

TABLE A-6. - Crystallization

Stream	Li	K	Na	Ca	SO_4	CO_3	H_2O	Total
Wt, g:								
13.....	23.62	670.64	548.23	0.14	1,969.54	101.44	7,709.55	11,023.1
15.....	1.36	427.23	419.44	.086	1,401.18	5.96	2,491.85	4,747
16.....	22.26	243.41	128.79	.054	568.36	95.48	5,217.7	6,276
wt pct:								
15.....	.03	9	8.84	.002	29.52	.12	52.49	100
g/L:								
13.....	2.74	77.8	63.6	.016	228.48	11.76	NAp	NAp[1]
16.....	4.11	45	23.81	.01	105.06	17.65	NAp	NAp[2]

NAp Not applicable.
[1]The total volume of stream 13 was 8.62 L solution with a specific gravity of 1.279.
[2]The total volume of stream 16 was 5.41 L solution with a specific gravity of 1.16.

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APPENDIX B.--PROCESS ECONOMICS

By D. A. Kramer[1]

PROCESS DESCRIPTION

The proposed process is designed to recover lithium, in the form of Li_2CO_3, from McDermitt clay. As-mined clay is crushed to minus 3/16-in and mixed with minus 3/16-in limestone and gypsum in a 5:3:3 ratio (clay-limestone-gypsum). These three materials are ground to minus 100 mesh, pelletized with water, and dried. Dried pellets are roasted at 900° C to react the gypsum with the lithium, producing lithium sulfate. Leaching the roasted pellets with water at ambient temperature extracts the soluble lithium sulfate and any other soluble sulfates formed during roasting. The leach slurry is filtered to separate the insoluble residue from the leachate.

Leachate is concentrated from 3 g/L Li to 13 g/L Li by evaporation, precipitating calcium impurities as a carbonate at the same time; these are separated from the solution by filtration. Mixing the concentrated solution with soda ash at 95° C precipitates lithium as Li_2CO_3. This slurry is filtered, and the solids are washed and dried to yield a Li_2CO_3 product. Total recovery is about 83 pct of the lithium in the clay.

After lithium precipitation, the remaining solution is cooled to 0° C to crystallize Na_2SO_4 and K_2SO_4 impurities that are leached from the clay. The crystal slurry is filtered, the crystals are discarded, and the mother liquor is recycled to the evaporator.

The proposed plant is designed to process 1,000 ton/d of McDermitt clay with the composition shown in table B-1. The material balance for the proposed process is detailed in figures B-1 through B-6. (Units are tons per day.)

[1]Chemist, Avondale Research Center, Bureau of Mines, Avondale, MD (now commodity specialist, Bureau of Mines, Washington, DC).

TABLE B-1. - Composition of McDermitt clay (dry basis), weight percent

Li	0.6
K	3.7
Na	.6
Mg	9
Fe	2.5
Ca	1.8
Al	3.1
F	2
SiO_2	53
Other	23.7
Total	100.0

ECONOMICS

Estimated capital and operating costs are based on the preceding process description.

Capital Costs

The capital cost estimate is of the general type called a study estimate by Weaver and Bauman.[2] This type of estimate, prepared from a flowsheet and a minimum of equipment data, can be expected to be within 30 pct of the actual plant cost.

The estimated capital cost on a second quarter 1984 basis (Marshall and Swift [M and S] index of 781.7) for a plant processing 1,000 ton/d of McDermitt clay is about $106 million, as shown in table B-2. Most of the plant is designed to operate 3 shifts per day, 7 days per week, 330 d/yr. Some of the clay preparation facilities only operate 1 shift per day, 7 days per week. The remainder of the time is for scheduled and unscheduled downtime.

[2]Weaver, J. B., and H. C. Bauman. Cost and Profitability Estimation. Sec. 25 in Perry's Chemical Engineers' Handbook, ed. by R. H. Perry and C. H. Chilton. McGraw-Hill, 5th ed., 1973, p. 47.

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TABLE B-2. - Estimated capital cost[1]

Fixed capital:	
Feed preparation section	$15,570,700
Roasting section	9,316,500
Leaching section	20,250,400
Evaporation section	9,347,300
Lithium recovery section	2,081,900
Crystallization section	3,420,800
Tailings pond	1,701,400
Steamplant	406,800
Subtotal	62,095,800
Plant facilities, 10 pct of above subtotal	6,209,600
Plant utilities, 12 pct of above subtotal	7,451,500
Basic plant cost	75,756,900
Escalation costs during construction	6,112,100
Total plant cost	81,869,000
Land cost	0
Subtotal	81,869,000
Interest during construction period	14,095,800
Fixed capital cost	95,964,800
Working capital:	
Raw material and supplies	1,081,500
Product and in-process inventory	2,993,800
Accounts receivable	2,993,800
Available cash	2,302,300
Working capital cost	9,371,400
Capitalized startup costs	959,600
Subtotal	10,331,000
Total capital cost	106,295,800

[1]Basis: M and S equipment cost index of 781.7.

Equipment costs used in this estimate are based on informal cost quotations from equipment manufacturers and on capacity-cost data. Cost data are brought up to date by the use of inflation indexes. In developing the plant capital costs, corrosion-resistant materials of construction were used where appropriate. For example, the tanks are rubber-lined to withstand the corrosive properties of the sulfate solution. The tailings pond is designed with a 2-yr capacity. It is assumed that after 2 yr the mine site will be developed to dispose of the remainder of the processing residue as backfill.

Factors for piping, etc., except for the foundation and electrical factors, are assigned to each section, using as a basis the effect fluids, solids, or a combination of fluids and solids may have on the process equipment. The foundation factor is estimated for each piece of equipment individually, and a factor for the entire section is calculated from the totals. The electrical factor is based on the motor horsepower requirements for each section. A factor of 10 pct, referred to as miscellaneous, is added to each section to cover minor equipment and construction costs that are not shown with the equipment listed.

For each section, the field indirect cost, which covers field supervision, inspection, temporary construction, equipment rental, and payroll overhead, is estimated at 10 pct of the direct cost. Engineering cost is estimated at 10 pct, and administration and overhead cost is estimated at 5 pct of the construction

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cost. A contingency allowance of 15 pct and a contractor's fee of 5 pct are included in the section costs.

The costs of plant facilities and plant utilities are estimated as 10 and 12 pct, respectively, of the total process section costs and include the same field indirect costs, engineering, administration and overhead, contingency allowance, and contractor's fee as are included in the section costs. Included under plant facilities are the cost of material and labor for auxiliary buildings such as offices, shops, laboratories, and cafeterias, and the cost of nonprocess equipment such as office furniture and safety, shop, and laboratory equipment. Also included are labor and material costs for site preparation such as clearing, grading, drainage, roads, and fences. The costs of water, power, and steam distribution systems are included under plant utilities. Cost for the plant owner's supervision is not included in the capital cost of the proposed plant.

Working capital is defined as the funds in addition to fixed capital, land investment, and startup costs that must be provided to operate the plant. Working capital, shown in table B-2, is estimated from the following items: (1) Raw material and supplies inventory (cost of raw material and operating supplies for 30 days), (2) product and in-process inventory (total operating cost for 30 days), (3) accounts receivable (total operating cost for 30 days), and (4) available cash (direct expenses for 30 days).

Capitalized startup costs are estimated as 1 pct of the fixed capital costs. Land investment is not included in this estimate.

Operating Costs

The estimated operating costs (table B-3) are based on an average of 330 days of operation per year over the life of the plant. The operating costs are divided into direct, indirect, and fixed costs.

Direct costs include raw materials, utilities, direct labor, plant maintenance, payroll overhead, and operating supplies. Raw materials and utility requirements per pound of Li_2CO_3 are shown in table B-4. The direct labor cost is estimated on the basis of assigning 4.2 employees for each position that operates 24 h/d, 7 days per week and 1.4 employees for each position that operates 8 h/d, 7 days per week. The cost of labor supervision is estimated as 15 pct of the labor cost.

Plant maintenance is separately estimated for each piece of equipment and for the buildings, electrical system, piping, plant utility distribution systems, and plant facilities.

Payroll overhead, estimated as 35 pct of direct labor and maintenance labor, includes vacation, sick leave, social security, and fringe benefits. The cost of operating supplies is estimated as 20 pct of the cost of plant maintenance.

Indirect costs are estimated as 40 pct of the direct labor and maintenance costs. The indirect costs include the expenses of control laboratories, accounting, plant protection and safety, plant administration, marketing, and company overhead. Research and overall administrative costs outside the plant are not included.

Fixed costs include the cost of taxes (excluding income taxes), insurance, and depreciation. The annual costs of both taxes and insurance are each estimated as 1 pct of the plant construction cost. Depreciation is based on a straight-line, 20-yr period.

The estimated annual operating cost of a plant processing 1,000 ton/d of McDermitt clay is about $36 million, as shown in table B-3. This corresponds to a cost of about $2.12/lb Li_2CO_3. No charge has been included for the clay.

The estimated operating cost of $2.12/lb Li_2CO_3 is about 1-1/2 times the current selling price of $1.48/lb.

ECONOMIC EVALUATION

After examining the high-cost areas in the proposed process, it appears that the largest cost factors are the limestone and gypsum costs. These costs contribute over 30 pct to the total operating cost. Raw material costs are high because of

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TABLE B-3. - Estimated annual operating cost

	Annual cost	Cost per pound of Li_2CO_3
Direct cost:		
McDermitt clay at $0.00/ton	$0	$0.0
Limestone at $26.50/ton	5,771,700	.366
Gypsum at $25.50/ton	5,225,700	.305
Soda ash at $90/ton	1,277,100	.074
Replacement balls for grinding at $0.27/lb	143,400	.008
Chemicals for steamplant water treatment	5,900	.001
Total	12,423,800	.724
Utilities:		
Electric power at $0.03/kW·h	793,000	.046
Process water at $0.25/Mgal	75,400	.004
Heavy oil at $0.80/gal	7,892,400	.460
Total	8,761,700	.510
Direct labor:		
Labor at $9/h	1,104,500	.064
Supervision, 15 pct of labor	165,700	.010
Total	1,270,200	.074
Plant maintenance:		
Labor	1,670,200	.097
Supervision, 20 pct of maintenance labor	334,000	.019
Materials	1,670,000	.097
Total	3,674,400	.213
Payroll overhead, 35 pct of above payroll	1,146,000	.067
Operating supplies, 20 pct of plant maintenance	734,900	.043
Total direct cost	28,011,000	1.631
Indirect cost, 40 pct of direct labor and maintenance	1,977,800	.115
Fixed cost:		
Taxes, 1 pct of total plant cost	818,700	.048
Insurance, 1 pct of total plant cost	818,700	.048
Depreciation, 20-yr life	4,798,200	.280
Total operating cost	36,424,400	2.122

TABLE B-4. - Raw material and utility requirements per pound of Li_2CO_3

Raw materials:		
McDermitt clay	ton	0.020
Limestone	ton	.013
Gypsum	ton	.012
Soda ash	ton	.001
Replacement balls for grinding	lb	.031
Utilities:		
Electric power	kW·h	1.542
Process water	Mgal	.018
Heavy oil	gal	.575

the plant location. It has been assumed that the quantity of limestone needed would be available from a deposit in Lyon County, NV, and that the required quantity of gypsum would be available from deposits in either Pershing or Washoe County in Nevada. Shipping charges from these distances have been included in the costs of these raw materials. These shipping charges significantly increase the costs of what would otherwise be relatively inexpensive raw materials.

To minimize the impact of these costs, the ratios of limestone and gypsum to the

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clay must be minimized without affecting lithium extraction. This would also aid in reducing the fuel requirements for roasting.

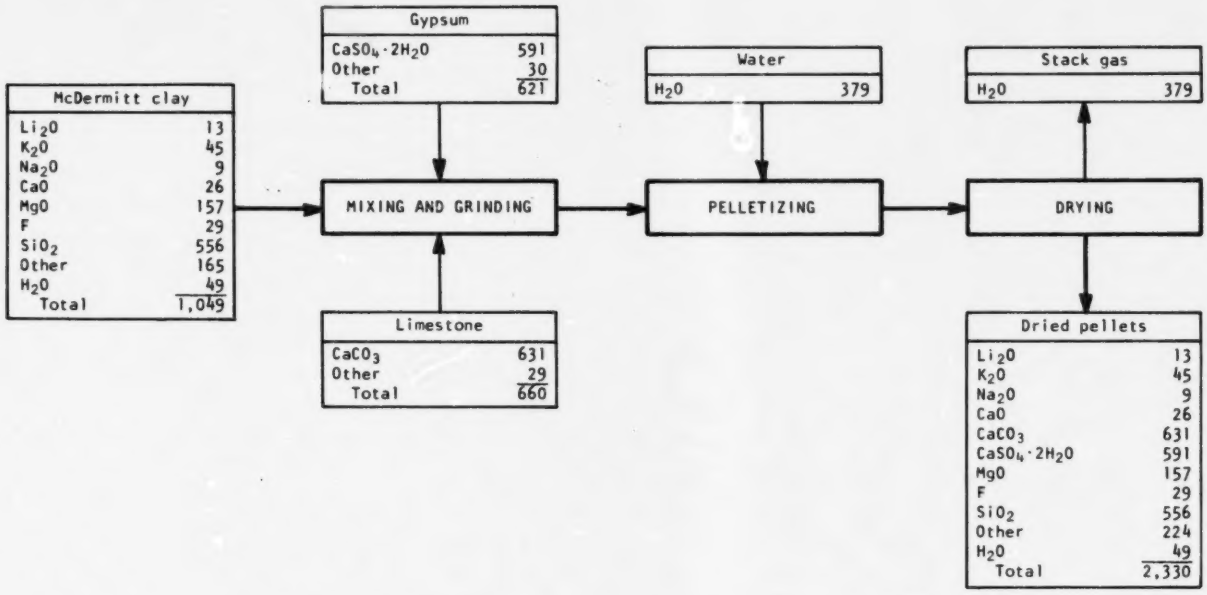

FIGURE B-1. - Feed preparation section (tons per day).

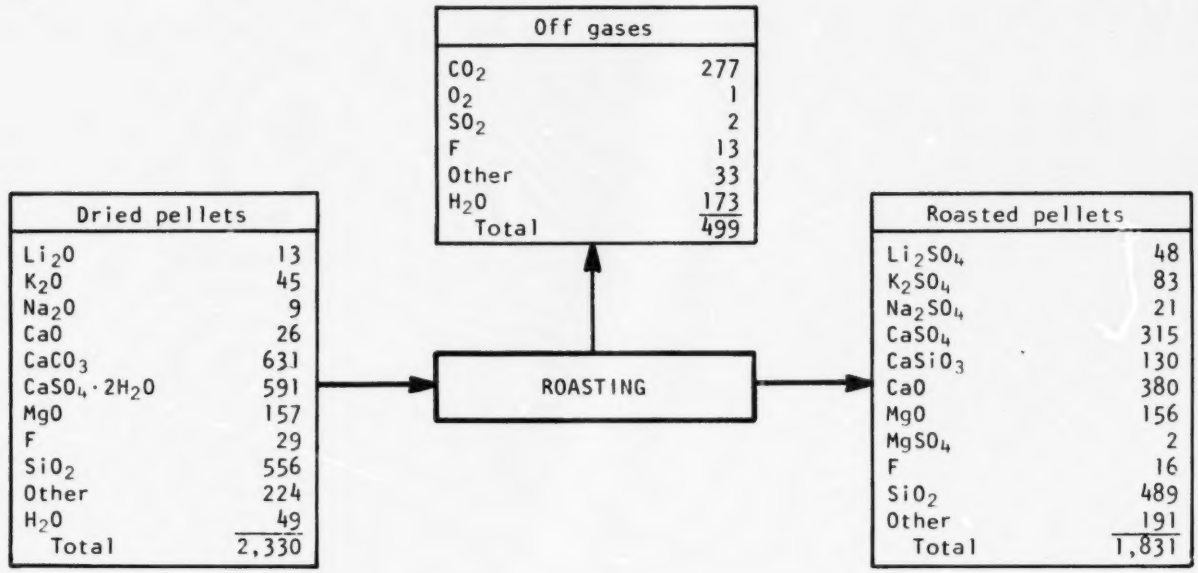

FIGURE B-2. - Roasting section (tons per day).

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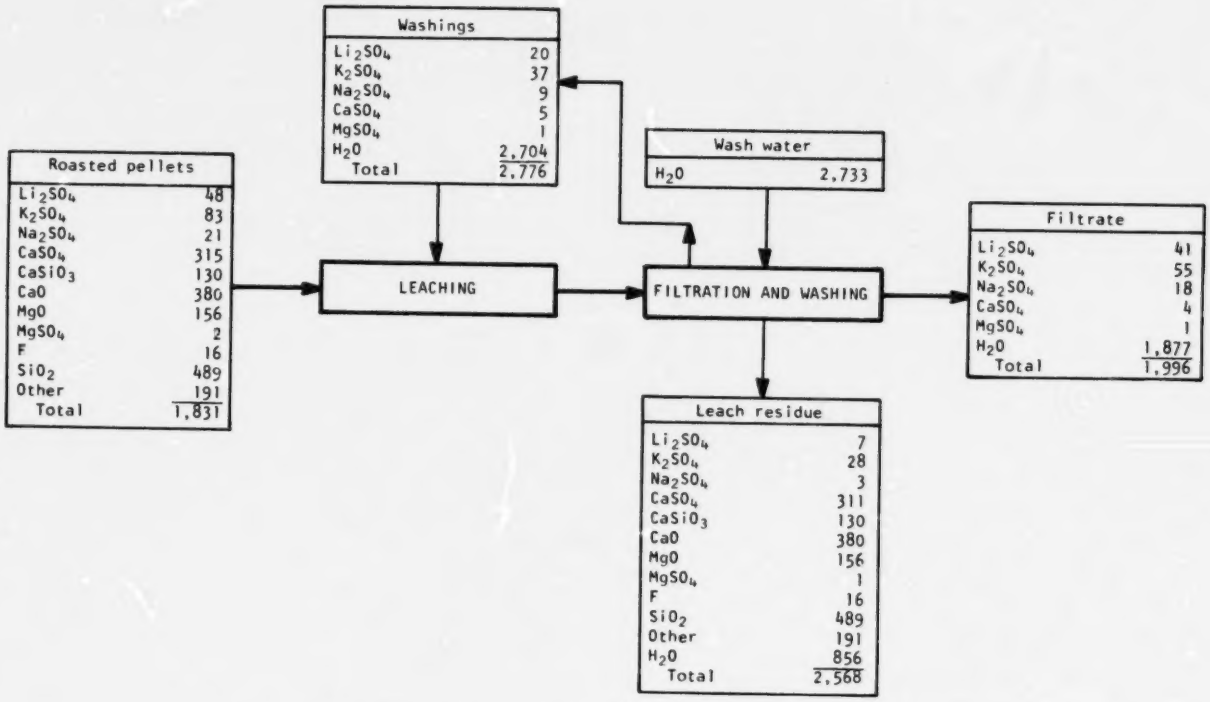

FIGURE B-3. - Leaching section (tons per day).

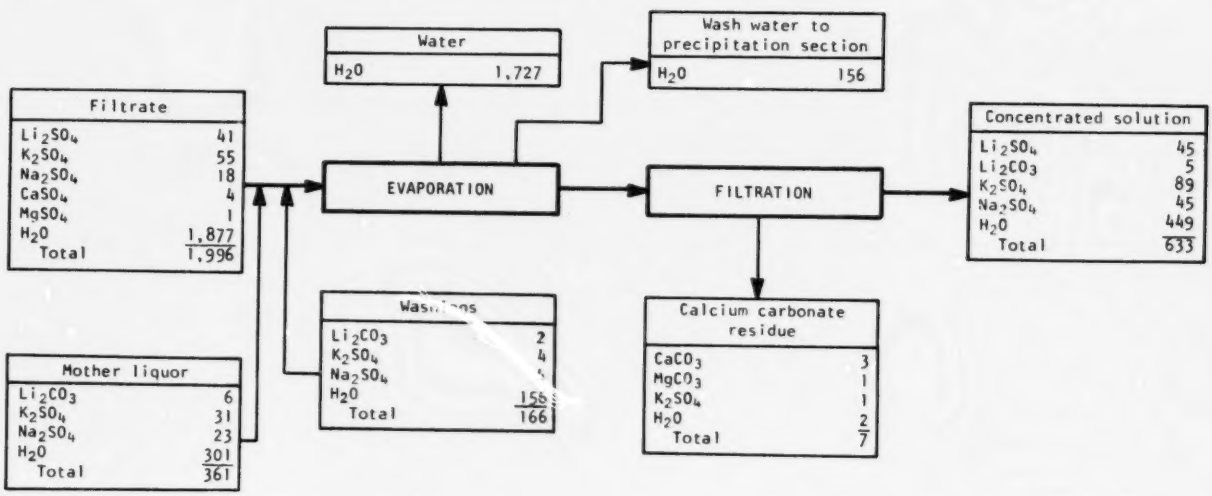

FIGURE B-4. - Evaporation section (tons per day).

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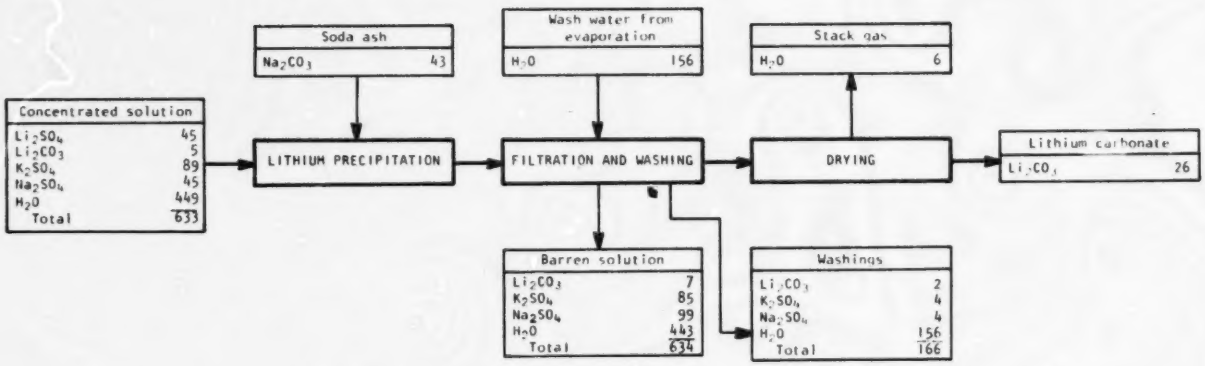

FIGURE B-5. - Lithium recovery section (tons per day).

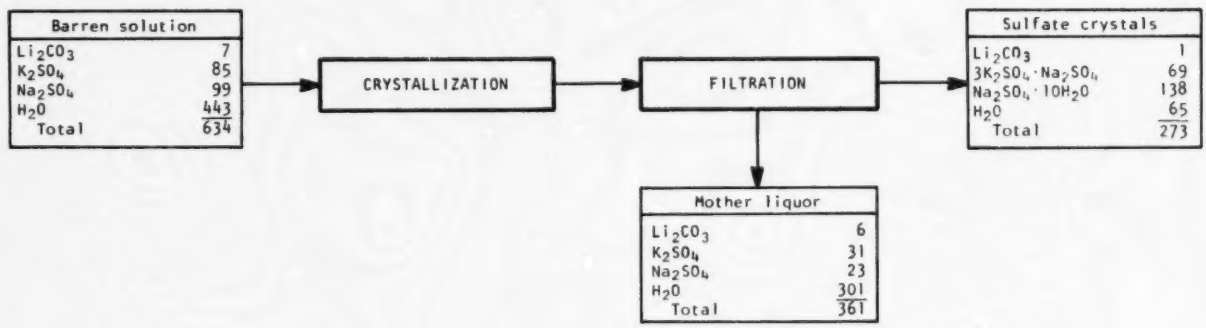

FIGURE B-6. - Crystallization section (tons per day).

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